

DOE/PC/91040-60

**ADVANCED DIRECT LIQUEFACTION CONCEPTS
for PETC GENERIC UNITS
Phase II**

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**Quarterly Technical Progress Report
for Period October through December 1995**

by

**University of Kentucky
Center for Applied Energy Research**

CONSOL Inc.

Hydrocarbon Technologies, Inc.

LDP Associates

February 1996

only that.

**Prepared for
The U. S. Department of Energy
Under Contract No.
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SUMMARY

TASK 1 MANAGEMENT PLAN

A detailed work plan for the first 15 months of the project was prepared and submitted that contains a discussion of activities that would occur during the 15 months of the project. The work plan is divided by tasks which includes laboratory support (Task 2), continuous bench-scale runs (Task 3), conceptual process design (Task 4) and preparation of an operations plan for a PDU facility (Task 5). In general, the format of the plan describes the work that will be performed under each task. Responsibilities associated with completing the work for Task is broken down by participant. A schedule including the time frame during which the individual work elements will be performed is also included along with a list of milestones associated with those activities. Progress by each participant will be monitored relative to this schedule and completion of the milestones.

TASK 2.1 LABORATORY SUPPORT (CAER)

The objective of the work at the CAER is to develop slurry catalysts that are sufficiently active to produce, in an all slurry mode, a product slate that is equivalent, from an economic standpoint, to that produced in the Wilsonville 263J base case. This will require an exceptionally active catalyst that can achieve in an all slurry mode a product distribution comparable to that was produced at Wilsonville in a 2-stage configuration. Initially, the work will be directed toward catalysts that are introduced into the reactor by impregnating the precursors onto the feed coal. In Phase I, we found that impregnated precursors could provide very reactive systems.

Catalyst Screening Test. The first step in this project is to develop a catalyst screening test (CST) by which catalyst candidates can be ranked relative to the base case catalyst. The CST will simulate liquefaction performance in an all-slurry liquefaction mode and will be standardized against an active catalyst that will be isolated from the ashy resid product from Wilsonville Run 262E. In Phase I, this Mo catalyst appeared to be far more active than any others that were tested. In the test, distillate

yield and coal conversion will be used as measures of catalyst activity.

The CST will be run in batch microautoclaves using techniques, similar to, but somewhat more severe than those used for the Phase I 1st-stage test in order to give higher resid conversion and hydrogen consumption. The same Wyodak coal is being used along with Wilsonville derived materials that are free of any solids. Specifically, the solvent is a solids-free mixture comprising 33 wt % Run 258B V-1074 heavy distillate and 67 wt % Run 258A V-130 ROSE deashed resid. In the CST a 524 °C (975 °F) cut point will be used to simulate that used at HTI in the continuous runs. The test will be run at 440 °C and a residence time that will be determined for the base case catalyst. Various pretreatment techniques will be used including a H₂ feed containing 3% H₂S. The workup technique will be essentially the same as used in Phase I.

In order to achieve a level of conversion in the CST that is consistent with the level of conversion observed in the base case Wilsonville Run 263J, a series of runs were made at different residence times with the Black Thunder coal in Wilsonville Run 262E solvent. Based upon these results, a residence time of 30 min was chosen. At this residence time a resid conversion of 44.2% was obtained versus a corresponding resid conversion of 37.5% in the base case. The corresponding resid conversion on maf coal in the base case was 85% versus the observed 92% in the CST. Therefore, the CST appears to be slightly more severe than obtained in Wilsonville Run 263J.

During this reporting period, the activity of the base case catalyst prepared by pressure filtration of the Wilsonville Run 262E V-1082 ashy resid was determined and compared with the conversion of coal in the absence of any added catalyst. This material was found to contain 740 mg Mo/kg, which is very close to values calculated from Mo concentration measurements that had been made on the original V-1082 ashy resid. In the CST, the pressure filtered solids that had been added to the reaction mixture to a level equivalent to the solids contained in Wilsonville Run 263J gave coal conversion of 98.2% with a resid conversion of 24%. The corresponding resid conversion on maf coal was 57%. Although this was considerably greater than the 19% resid conversion observed in the absence of any catalyst (41% based on maf coal), it was far short of the anticipated 44.2% resid

conversion (92% resid conversion on maf coal) observed in the base case run. We are currently investigating why we were unable to achieve the anticipated level of conversion in the CST with the standard catalyst. Interestingly, the run in the absence of any catalyst gave a THF coal conversion of 68%, which is reasonable agreement to the 76.2% value obtained previously after 22 min in a Run 262E solids-free, resid-containing solvent.

Catalyst activation studies on metal impregnated catalysts were performed during this reporting period. The experimental work is initially concentrating on determining the effect of presulfiding conditions on activating a Mo-impregnated coal with different H_2S/H_2 mixtures at different temperatures and reaction times. Mo, Mo + Ni, and Mo + Fe metal impregnated Black Thunder Wyodak coals were prepared by an incipient-wetness method with aqueous solutions of ammonium molybdate, nickel sulfate and ferrous sulfate. Each of the metal-impregnated coals were tested in the CST solvent in microautoclaves for 60 min in 3% H_2S/H_2 at 1000 psig (cold) and 440 °C. Comparative runs without catalyst and with only Molyvan L were also made. The resid conversion numbers ranged from 36.8 to 40.8% while the non-catalyzed run gave only 21.8%. THF coal conversions for the metal-impregnated coals were in excess of 90% in every case compared to 66.3% in the non-catalyzed case. The resid conversion in the base case solvent with 30% coal after 60 min was observed to be 47%, which is higher than observed for these catalysts. A Mo-impregnated coal that had been subjected to a pretreatment at 375 °C in a H_2S/H_2 stream gave a slightly smaller resid conversion than the non-pretreated case.

TASK 2.2 LABORATORY SUPPORT (CONSOL)

No activity.

TASK 3 CONTINUOUS OPERATIONS/PARAMETRIC STUDIES

TASK 3.3 (HYDROCARBON TECHNOLOGIES, INC.)

A 200 pound composite sample of pressure filter liquid from previous bench runs with dispersed catalyst and Black Thunder coal was distilled to remove low boiling materials and vacuum distilled to generate a 524 °C- (975 °F-) vacuum still overhead (VSOH). The VSOH contains only 5.15%

wax. Various portions of this sample will be hydrodewaxed and used in preparing start-up/make-up solvent in Condition 5 of ALC-1. Based upon subsequent discussions, a sample will also be shipped to Sandia National Lab for hydrotreating

The following samples were provided:

Bulk samples of Black Thunder coal were sent to CAER and CONSOL as well as a 20 pound sample of pulverized Black Thunder coal and 250 gram sample of HTI Fe-based catalyst were forwarded to CAER.

TASK 4 CONCEPTUAL PROCESS DESIGN

Task 4.1 PROCESS MODELING

Prof. Eric Grulke from the Chemical Engineering Department will be developing an ASPEN model of the liquefaction process in conjunction with Mike Peluso from LDP Associates. Discussions were held on the makeup of the model and sources of feed and product characterization data.

TASK 4.4 PRELIMINARY TECHNICAL ASSESSMENT (LDP ASSOCIATES)

The computerized modeling of the overall liquefaction, gasification and upgrading systems was completed. The modeling includes elementally balanced, material balances for all the systems as well as a determination of the interrelated hydrogen and fuel gas balances. Individual process unit simulations provide most of the input data required by the model.

It was determined that the previously assumed coal and ash concentrate gasification temperature was significantly higher (2,920°F vs 2,500°F) than necessary to achieve slagging of the ash. A more accurate C₁ to C₃ gas yield distribution in the upgrading units (hydrotreating, hydrocracking and catalytic reforming) was determined based on experimental data contained in a Chevron report. The Base case is being updated to incorporate the above revisions.

Economic projections are being prepared for each of the four advanced concepts to be tested in the first continuous bench scale run (ALC-1) at HTI. The implications of these projections will be discussed at the ALC-1 Run Plan Meeting to be held on January 19.

Preliminary data from Exxon's second bench scale run was obtained. Dispersed moly was the only catalyst used for most of the run. The run data indicate that both higher hydrogen treat gas rate and a higher moly catalyst makeup rate (100 vs 50 ppm) increased distillate yield. However, increasing the moly catalyst makeup rate to 750 ppm from 100 ppm had no effect on distillate yield.

A meeting was held in Lexington with Professor Eric Grulke of the University of Kentucky in an effort to define areas where process modeling using the ASPEN simulator might be useful. Grulke was briefed on similar work done by AMOCO/Bechtel and ICRC and given hard copy reference material.

SECTION ONE

**Center for Applied Energy Research
University of Kentucky**

TASK 2.1 LABORATORY SUPPORT (UK/CAER)

Introduction

The objective of the work that will be conducted at the CAER under this project in Phase II is to develop viable slurry catalysts for producing a distillate product equivalent to the Wilsonville 263J base case, in which Wyodak coal from the Black Thunder mine was used as feed. Unlike the base case run which was made in a 2-stage configuration in which the 1st-stage was operated as a thermal reactor with a Mo-Fe dispersed catalyst, and a 2nd-stage ebullated bed reactor with a Ni-Mo extrudate, this project is switching to an all-slurry reactor configuration using only dispersed catalyst. Through elimination of the 2nd-stage catalyst, considerable cost reduction could be achieved if a dispersed catalyst can be found that can achieve the same level of coal conversion and product selectivity that was obtained in the 2-stage Wilsonville operation. This requires that the process operate with the same thermal efficiency while consuming the same or lower amounts of H_2 .

Various methods of introducing the dispersed catalyst precursors into the reaction system that can provide a significant reduction in catalyst cost relative to the catalysts used in the base case Wilsonville run will be investigated in this project. In Phase I, we found that impregnating the catalytic metal precursors onto coal and activating these metals *in situ* provided very reactive systems. To use these catalyst systems requires that several interrelated process parameters be optimized in order to generate active catalysts. Although Mo is an active component in all these catalysts, the co-metals and their particular salt precursors must be defined as well as the concentration of metal on the coal substrate. In addition, limitations on the amount of the feed coal that must be impregnated, the conditions and reactants necessary to activate the catalyst as well as maintain its activity in the process, and the concentration of the catalyst in the recycle stream must be specified. The effect of each catalyst on product yields and quality must also be determined. Therefore, as considerable amount of work is necessary before the specific catalysts can be selected for the individual bench-scale runs.

Task 2.1.1 Development of a Catalyst Screening Test (CST)

A catalyst screening test is being developed to facilitate the selection of catalysts and their concentrations in the reaction system. This test will simulate liquefaction performance in an all-slurry liquefaction mode and test expands upon the catalyst screening tests used in Phase I to simulate the first-stage reactor. An active catalyst, isolated from the ashy resid product from Wilsonville run 262E, is being used as a reference material, against which the performance of the test materials will be assessed. This Mo catalyst appeared to be far more active than any others tested in Phase I. Distillate yield and coal conversion will be used as the measure of catalyst activity. The primary objective is to reproduce the coal and resid conversions obtained in Wilsonville Run 263, in which only distillate product was achieved, but at significantly lower projected operating costs. The catalysts that will be examined, Mo, Mo+Ni, and Mo+Fe formulations, will be added to the coal by impregnation and the tests will be conducted at the selected CST conditions.

CST Test Conditions. The CST will be run in batch microautoclaves using techniques, similar to, but somewhat more severe than those used for the 1st-stage tests. In the CST, resid conversion and corresponding hydrogen consumption will be significantly higher than in the previous test. The amount of H_2 in the reactor, excluding the stem to the pressure transducer, in the CST, is approximately 15 wt % of the dry feed coal. Total hydrogen, which includes that in the line leading to the pressure transducer, is about 18 wt % of dry coal. In runs made thus far, a nominal 1350 psig initial pressure at ambient temperature has been used which results in a pressure of approximately 2500 psig at operating pressure.

As in the Phase I test, the same coal and Wilsonville derived solvent is used. The coal is a Wyodak coal from the Black Thunder mine and the solvent is a solids-free mixture comprising 33 wt % Run 258B V-1074 heavy distillate and 67 wt % Run 258A V-130 ROSE deashed resid. Although the deashed resid was a bottoms from a 565 °C (1050 °F) vacuum tower, it still contained 24.3% 565 °C- (1050 °F-) material and 8.3% 524 °C- (975 °F-) material. Since the cut point has been changed to 524 °C from 565 °C, in order to conform to the cut point that will be used at HTI in the continuous runs, the composition of the starting material differs from the weight ratio of these two materials.

To determine the amount of 524 °C distillate and resid in the reaction feed, a sample of the deashed resid alone and the mixture of solvent, comprising deashed resid and heavy distillate, were distilled to 295 °C at 1 mm Hg (for an AET of 524 °C) in the usual distillation apparatus. The results of those distillations are shown in Table 1.

Table 1. Distillation of reaction feeds to a 524 °C atmospheric equivalent temperature end point.		
	Wt% maf deashed resid	Wt% maf solvent ^a
524 °C- distillate	8.3	40.9
524 °C+ resid	91.7	59.1
Total	100.0	100.0
a. A mixture of 67.4% deashed resid and 32.6% heavy distillate from Wilsonville R-258A.		

The ash content of the deashed resid was determined to be 0.13 wt% and the distillate 0.12 wt%.

Directionally, we would expect that the amount of distillate would decrease and the amount of resid to increase. Actually, we found the opposite occurred. The 524 °C concentration in distilled Run 258B V-1074 distillate was found to be 3% and in the Run 258A deashed resid was 8.3%. This gives a computed composition for the mixture in of 38% distillate and 62% resid. This mixture distilled to a 524 °C- cut point gave 41% distillate and 59% resid, as shown in Table 1, which is reasonably close to the computed values. The 41 wt % 524 °C- distillate in the mix was actually higher than the concentration of the V-1074 heavy distillate (33 wt %) that was used to prepare the mix. This merely indicates that the vacuum tower at Wilsonville contained considerable 565 °C material in the distillation bottoms.

The feed composition in the CST is shown in Table 2. The coal concentration in the mix remains the same. However, the split of distillate and resid changes with the resid concentration decreasing while the distillate fraction increases.

Table 2. Composition of Feed to Catalyst Screening Test (CST)				
	As-received coal basis, wt %	Dry coal basis, wt %	Feed based on 524 °C cut point, dry basis, wt %	Base case, Dry coal basis, wt %
Cut point, °F	565	565	524	565
Dry coal	32.3	35.3	35	35.0
Run 258 B Distillate	19.5	21.3	25	31.9
Run 258A Deashed Resid	39.6	43.4	40	33.1
Moisture	8.6			
Total	100	100	100	100

The test will be run at a temperature of 440 °C and a residence time that will be determined for the base case catalyst. The gas will differ from the Phase I test in that it will contain 3% H₂S, whereas in the Phase I test DMDS was added to sulfide the iron catalyst.

Experimental Test Method. In the CST test, the microautoclave is loaded with solvent, coal (+ catalyst) and sulfiding agent, if necessary, before being pressurized with approximately 1350 psig of cold 3% H₂S in hydrogen and leak tested. Any special catalyst activation pretreatment will be carried out at this stage as specified per data developed during the development of the catalyst. Otherwise, the reactor will then be immersed in a heated, fluidized sandbath and continuously agitated at a rate of 300 cycles per minute for the specified residence time. At the end of the reaction period, the reactor will be quenched and cooled to ambient temperature before the gaseous products are collected and sampled for analysis by gas chromatography. The solid and liquid products will be removed from the reactor using THF and the mixture extracted in a Soxhlet extraction apparatus for 18 hours. The THF insoluble material will be dried (80 °C at 125 torr) and weighed. The soluble fraction is concentrated by removing excess THF in a rotary evaporator and subjected to vacuum distillation using a modified ASTM D-1160 procedure. The distillate will be analyzed by simulated

$$\text{Resid Conv} = 100 \left[1 - \frac{[\text{IOM} + 524^{\circ}\text{C} \cdot \text{Resid (maf)}]_{\text{Products}}}{[\text{Coal (maf)} + \text{IOM} + 524^{\circ}\text{C} \cdot \text{Resid (maf)}]_{\text{Feed}}} \right]$$

distillation. Material balances are based upon a forced ash balance in which distillate is calculated by difference and includes water and any experimental error. Product distributions are calculated by subtracting the weights of gases, residue, ash and IOM in the product from the weight of each in the feed. Resid conversions are calculated as shown in Equation 1, while coal conversion is derived from the net yield of IOM. Determination of the CST Reaction Time. In order to achieve a level of conversion in the CST that is consistent with the level of conversion observed in the base case Wilsonville Run 263J, a series of runs were made at different residence times with the Black Thunder coal in Wilsonville Run 262E solvent, which is equivalent to the Wilsonville 263J recycle solvent. Based upon those results, it appears that 30 min residence time will provide a level of resid conversion consistent with the base case. Those data are shown in Table 3.

At 30 min in the CST, resid conversion was 44.2% versus a corresponding resid conversion of 37.5% in the base case. The corresponding resid conversion on maf coal in the base case was 85% versus the observed 92% in the CST. The resid conversion is slightly higher than in the base case, which was 37.5% (85% on maf coal).

A tentative value of 47% for resid conversion in the base case system after 60 min was obtained under CST conditions. This value departed significantly from a linear plot of log unconverted resid versus time, whereas the 22 and 30 minute data fell on a linear plot (see Figure 1).

Activity of Base Case Catalyst. The activity of the base case catalyst in the CST was determined and compared with the conversion of coal in the absence of any added catalyst. The reference catalyst was prepared both by pressure filtration as well as by Soxhlet extraction. The filtration was performed

Figure 1

Resid Conversion in 262E V-131B

440 C, 1400 psi (cold) 2% H₂S/H₂, 30% coal

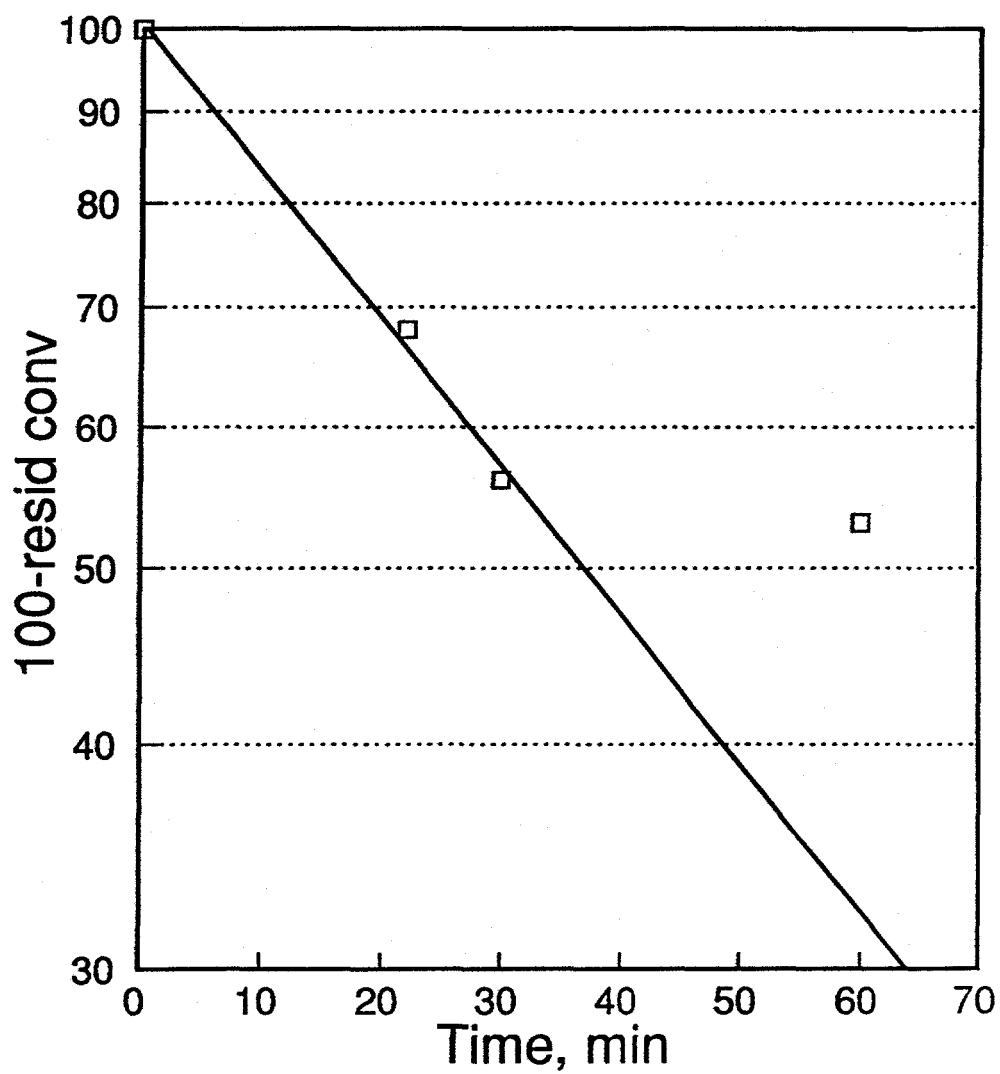


Table 3. Black Thunder liquefaction in WV Run 262 Recycle Solvent		
Run No.	TS-339-1 TS-340-1 TS-341-1	TS-310-1 TS-311-1
Feed Composition		
Coal, mf, wt%	30.0	
Heavy Distillate (V-1074), wt%	30.5	
Ashy Resid (V-1082), wt%	39.5	
Added Catalyst, wt%	none	
Mo Concentration, mg/kg mf coal	380	
Conditions		
Temperature	440 °C	
H ₂ pressure, ambient, psia	1300	
H ₂ S pressure, ambient, psia	26	
Time, min	30	60 ‡
Product		
Coal Conv	105.6	106
Gas Yield	18.3	27
Resid Conv	44.2	47
Resid Conv based on maf coal	92	98
H ₂ consumption, wt%	4.6	5.4
‡ Tentative data		

on a THF slurry of the Run 262E V-1082 ashy resid at ambient temperature and the Soxhlet extraction was done with THF at refluxing temperature. Both materials were dried overnight at 40 °C at 125 mm Hg. The filter cake obtained from the Run 262E V-1082 was found to contain

740 mg Mo/kg. Results for the pressure filtered material are reported here.

In the absence of any added catalyst, the composition of the starting slurry was 35% dry coal, 21.5% V-1074 565 °C- (1050 °F-) heavy distillate and 43.6% V-1082 deashed material. The composition on a 524 °C cut point basis was 35% dry coal, 25% 524 °C- distillate and 40% 524 °C+ resid. In the CST, as shown in Table 4, resid conversion was 20% while resid conversion, based on

Table 4. CST results in absence of added catalyst and with base case catalyst		
Run No.	TS-024-1 TS-032-1	TS-026-1 TS-025-1
Feed Composition		
Coal, mf, wt%	35.0	30.7
Run 262E Heavy Distillate (V-1074), wt%	21.5	18.8
Run 262E Deashed Resid (V-130), wt%	43.6	38.1
Catalyst, Run 262 V-1082 extract wt%	none	12.4
Mo Concentration, mg/kg mf coal	none	295
Conditions		
Temperature	440 °C	440 °C
Time, min	30	30
H ₂ pressure, ambient, psig	1204	1174
H ₂ S pressure, ambient, psig	37	36
Product		
Coal Conv	70	98.2
Gas Yield	18	18.2
Resid Conv	20	24
Resid Conv based on maf coal	44	57
H ₂ consumption, wt%	1.6	4.5

maf coal, was 44%. Both were relatively low. Overall conversion to THF solubles was only 70%, which is also surprisingly low. Previously, when this coal was liquefied in a mixture of heavy distillate and deashed resid from Run 262E under the Phase I test conditions for 22 min, a negative yield of pentane-soluble product was obtained and the corresponding conversion to THF solubles was 76.2%.¹

Upon adding the pressure filtered solids from Run 262E V-1082 ashy resid at a level equivalent to adding back the solids to the deashed resid, the conversion improved. Coal conversion went to 98.2% while resid conversion increased to 24%. The corresponding resid conversion on maf coal was 57%. Relative to the non-catalyzed run, conversions increased markedly, however not to the level observed in the base case solvent system presented in Table 3. The reason for this difference isn't understood at present. The change in cut point from 1050 to 975 °F could have some effect on this result and will be investigated.

Task 2.1.2 Activation of Impregnated Metal Catalysts

The results from Phase I showed that in the resid conversion test the Mo precursor that we added was not being converted into an active catalyst rapidly enough to provide the same level of resid conversion as recycled catalyst. Although it isn't known how long it took the Mo in the Wilsonville runs to become active, i.e., how many passes through the reactor, this apparently had little impact on overall plant performance since these runs were made at a high recycle rate. The recycle to fresh-catalyst addition rate used in Run 263J was 3.5/1. Operating at these levels of catalyst recycle, the failure of the freshly added Mo to activate immediately would have minimal impact on the operation. However, the continuous runs that will be made in this program will operate at a much lower catalyst recycle rate which increases the need to achieve catalyst activation in the first pass. Activation is even more important in the laboratory catalyst development work since activity is measured only on fresh catalyst. Therefore, the objective of this task is to determine how to activate the Mo catalysts in lab scale reactors in order to improve activity in the continuous runs as well as be able to effectively screen catalyst candidates in the laboratory.

The mechanism by which these catalysts are activated is not understood. It had been assumed that sulfiding the Mo precursors would convert these materials into their active form. Such is not the case, apparently, since our work as well as others have shown that adding sufficient sulfur to the system under conditions that are presumed severe enough to produce Mo sulfide does not always generate an active catalyst. The experimental work will initially concentrate on determining the effect of various parameters on activating the Mo catalysts. Specifically, the effect of presulfiding conditions on activating a Mo-impregnated coal will be determined by treating coal slurries with different H_2S/H_2 mixtures at different temperatures and reaction times.

During this reporting period, Mo, Mo + Ni, and Mo + Fe metal impregnated Black Thunder Wyodak coals were prepared by an incipient-wetness method with aqueous solutions of ammonium molybdate, nickel sulfate and ferrous sulfate. Approximately 20 g samples of each of the coals were then dried for 2.5 hours at 40 °C and 1 psi. The concentration of metal, on a dry coal basis, and the moisture content of the samples are shown in Table 5

Table 5. Metal impregnated Wyodak coals				
Coal sample no.	Mo, ppmw mf coal	Ni, ppmw mf coal	Fe, ppmw mf coal	Moisture, wt% as-determined
CB-51	552	-	-	30.1
CB-52	538	101	-	21.0
CB-53	478	-	6730	22.0

Each of these metal-impregnated coals were then tested in the CST solvent in microautoclaves for 60 min in 3% H_2S/H_2 at 1000 psig (cold) and 440 °C. Subsequent studies showed 30 minutes to be the reaction time needed to match the base case (R-263J) resid conversion for both reactor stages. Runs for 30 min residence times are planned. The results are compared with coal reaction in the absence of catalyst as well as in the presence of Molyvan L in Table 6. The Mo-only impregnated coal was also pretreated at 375 °C for 30 min in the H_2S/H_2 mixture.

In all these runs the resid conversion numbers for the metal impregnated coals were significantly higher than for the run in the absence of catalyst. For the metal-impregnated coals these resid conversions ranged from 36.8 to 40.8% (82-96% resid conv on maf coal) while the non-catalyzed run gave only 21.8%. THF coal conversions for the metal-impregnated coals were in excess of 90% in every case to compare with 66.3% in the non-catalyzed case. The yields of HC and CO+CO₂ gases in the thermal case exceeded the catalyzed cases by a small amount. The resid conversion in the base case solvent with 30% coal after 60 min was observed to be 47%, which was somewhat higher than observed for these catalysts. Since that is still a tentative value, we remain somewhat uncertain whether these catalysts have been activated.

Table 6. Results of 60 minute liquefaction experiments.						
Catalyst preparation	None	Molyvan L	CB-51		CB-52	CB-53
Metals added	-	Mo	Mo	Mo	Ni/Mo	Mo/Fe
Pretreat time at 375 °C, min	0	0	0	30	0	0
Products, wt% maf Coal						
HC Gases	21.5	21.0	19.7	20.7	17.9	17.2
CO+CO ₂	12.2	11.9	11.1	11.3	11.5	11.2
524 °C-	41.6	61.9	67.1	64.1	64.9	63.0
524 °C+	24.7	5.2	2.1	3.9	5.7	8.6
Total	100.0	100.0	100.0	100.0	100.0	100.0
THF Conv	66.3	95.1	90.6	90.6	92.3	92.5
Resid Conv, wt% maf resid	21.8	40.4	40.8	38.9	40.7	36.8
Resid Conv, wt % maf coal	49.3	89.6	95.5	91.2	89.1	82.6
Material Bal index	97.3	97.1	97.1	102.3	89.0	94.2
H ₂ consumed, mg/g maf Coal	37.5	64.7	70.1	89.4	70.3	65.6
Coal moisture, wt %	22.9	22.9	30.1	30.1	21.1	22.6
Run No.	B5-349-1	B5-347-1	B5-346-1	B5-353-1	B6-4-1	B6-10-1
a. Liquefaction experiments at 440°C for 60 minutes, using 3 g Black Thunder coal, 2.90 g V-130 deashed resid and 1.43 g V-1074 heavy distillate from Wilsonville Run 258A. 1350 psig cold charge pressure, including 3% H ₂ S. SO ₂ -free ash basis.						

The Mo-impregnated coal was also subjected to a pretreatment at 375 °C in the H₂S/H₂ stream. The resid conversion in this test was slightly smaller than the run made without pretreatment indicating no significant difference in resid conversion from the non-pretreated case.

References

1. Quarterly Report, April-June 1994, Advanced Direct Liquefaction Concepts for PETC Generic Units, U. S. Department of Energy No. DOE/PC/91040-45, September 1994, p 1-22.

SECTION TWO

CONSOL, INC

January 11, 1996

Report Period: Oct 1-Dec, 31, 1995

SUBCONTRACT TITLE AND NUMBER:

Subcontract UKRF-4-25582-92-76 to CONSOL Inc.
Under DOE Contract No. DE-AC22-91PC91040, "Advanced Coal Liquefaction
Concepts for the PETC Generic Bench-Scale Unit"

SUBCONTRACTOR NAME:

CONSOL Inc.
Research & Development
4000 Brownsville Road
Library, PA 15129

SUBCONTRACT PERIOD: November 26, 1991 - September 30, 1996

PRINCIPAL INVESTIGATORS: F. P. Burke, R. A. Winschel, G. A. Robbins

1. SUBCONTRACT OBJECTIVES:

To develop selected technology concepts that have the potential to reduce the cost of producing liquid fuels by direct coal liquefaction.

2. TECHNICAL APPROACH CHANGES:

The subcontract was modified to initiate Phase 2, in which promising concepts from Phase 1 will be evaluated in a continuous liquefaction unit to demonstrate the performance of the process concepts and to provide data for the technical and economic assessment.

3. CONTRACT TASKS:

Task 1 - Management Plan

The CONSOL contribution to the Management Plan for Phase II, including plans for Run ALC-1, the first of four planned bench-scale runs, were prepared, reviewed by the project participants, revised, and issued. Comments were supplied to CAER on the other section of the Management Plan. Our comments included suggested modification to the draft plans for Run ALC-2.

Task 2 - Laboratory Support

HTI sent 1200 lb (four drums) of pulverized Black Thunder Mine coal for agglomeration.

4. OPEN ITEMS: None

5. SUMMARY ASSESSMENT AND FORECAST:

Next month, the four drums of Black Thunder coal obtained from HTI will be sampled and analyzed. Laboratory-scale agglomeration tests will be conducted with the coal to verify its performance in preparation for the production of the feed agglomerated coal for Run ALC-1. Plans for Run ALC-1 will be further developed through discussions with the project participants.

A handwritten signature in black ink, appearing to read 'F. P. Burke', is written above the typed name.

F. P. Burke
Project Manager

/ls

SECTION THREE

HYDROCARBON TECHNOLOGIES, INC

Hydrocarbon Technologies, Inc.
P.O. Box 6047
New York & Puritan Avenues
Lawrenceville, New Jersey 08648
609/394-3102 Fax 609/394-9602



Theo L.K. Lee
Vice President

January 18, 1996

Dr. Ed Givens
University of Kentucky
Center for Applied Energy Research
3572 Iron Works Pike
Lexington, KY 40511-8433

RE: DOE Advanced Coal Liquefaction Concepts - Phase II Program
Subcontract: UKRF425582-96-152
Progress Report: December/95

Dear Ed:

In addition to the bulk coal samples sent to CAER and CONSOL, a smaller sample of pulverized Black Thunder coal (20 lb.) and 250 gm of HTI Fe-based catalyst (in form of wet cake) were also forwarded to CAER during November/December of last year.

A composite pressure filter liquid sample of 200 lb. from previous bench runs obtained from dispersed catalyst and Black Thunder coal has been prepared. This sample will serve as a pool for liquid required for the dewaxing work at CONSOL, for hydrogenation screening test at Sandia National Lab and for preparing start-up/make-up solvent (for Condition 5 of ALC-01). Distribution of the composite PFL will be implemented within the first two weeks of January, 1996.

Al Comolli, Dr. Jianli Hu and I will be attending the January 19th Pre-run meeting to be held at the Princeton Hyatt Hotel. Do you know who will be participating in the HTI facility tour on January 18th (Thursday)?

Sincerely yours,



Theo L.K. Lee

LKL/dms

cc: R. Winschel (CONSOL)
F. Stohl/J. Miller (Sandia)
M. Peluso (LDP)
J. Hu
A. Comolli

Hydrocarbon Technologies, Inc.
P.O. Box 6047
New York & Puritan Avenues
Lawrenceville, New Jersey 08648
609/394-3102 Fax: 609/394-9602



Theo L.K. Lee
Vice President

December 15, 1995

Via Fax No. (606) 257-0302

Dr. Ed. Givens
University of Kentucky
Center For Applied Energy Research
3572 Iron Works Pike
Lexington, KY 40511-8433

Dear Ed:

RE: PROGRESS REPORT FOR MONTH ENDING 10/31/95
DOE Advanced Concepts Program (Phase II)
DE-AC22-91PC91040

1. PROJECT MANAGEMENT

- Received and signed sub-contract document.

2. COAL AND CATALYST SAMPLES

- Shipped 5 gallons of pulverized Black Thunder Coal to CAER

3. UNIT AND SOLVENT PREPARATION

- No activity

cc: F. Derbyshire (CAER)
F. Stevens
J. Hu
V. Pradhan

Hydrocarbon Technologies, Inc.
P.O. Box 6047
New York & Puritan Avenues
Lawrenceville, New Jersey 08648
609/394-3102 Fax 609/394-9602



Theo L.K. Lee
Vice President

November 10, 1995

Dr. Ed Givens
University of Kentucky
Center for Applied Energy Research
3572 Iron Works Pike
Lexington, KY 40511-8433

Dear Ed:

RE: PROGRESS REPORT FOR MONTH ENDING 10/31/95
DOE Advanced Concepts Program (Phase II)
DE-AC22-91PC91040

1. PROJECT MANAGEMENT PLAN

- attended a kickoff meeting held at CAER to finalize the Project Management Plan (PMP).
- reviewed and commented on the draft PMP prepared by CAER and CONSOL.
- distributed the 11th Quarterly Report of HTI's DOE Catalytic Multistage Liquefaction Program during the kickoff meeting.

2. COAL AND CATALYST SAMPLES

- shipped 1200 lbs. of minus 2" Black Thunder Coal to CAER
- shipped 1200 lbs. of pulverized Black Thunder Coal to CONSOL
- shipped 25 g (dry basis) of HTI Fe based catalyst to CAER

3. SOLVENT INVENTORY

The following pressure filter liquid from various bench runs using Black Thunder Coal have been identified:

SECTION FOUR

LDP ASSOCIATES

LDP ASSOCIATES

Michael Peluso, Proprietor
609-586-2301

32 Albert E. Bonacci Dr.
Hamilton Square, N.J. 08690

January 24, 1996

Dr. Ed Givens
Center for Applied Energy Research
3572 Iron Works Pike
Lexington, Kentucky 40511-8433

Dear Ed:

Subject: QUARTERLY PROGRESS REPORT FOR OCT. THRU DEC. 1995

For the quarter ending December 31, 1995 the following subcontract services (UKRF-4-25582-92-75) were performed in support of the DOE Advanced Concepts Program (DE-AC22-91PC91040):

PROJECT MANAGEMENT PLAN

A kickoff meeting was held at CAER in October to discuss finalization of the Project Management Plan(PMP). A subsequent draft of the PMP was reviewed and comments were sent to CAER and CONSOL. A write-up for Task 4, Conceptual Process Design, was prepared.

ECONOMIC ASSESSMENT

The computerized modeling of the overall liquefaction, gasification and upgrading systems was completed. The modeling includes elementally balanced, material balances for all the systems as well as a determination of the interrelated hydrogen and fuel gas balances. Individual process unit simulations provide most of the input data required by the model.

It was determined that the previously assumed coal and ash concentrate gasification temperature was significantly higher (2,920°F vs 2,500°F) than that necessary to achieve slagging of the ash. In addition, a more accurate C₁ to C₃ gas yield distribution in the upgrading units (hydrotreating, hydrocracking & catalytic reforming) was determined based on experimental data contained in Chevron report DE82001127 (Refining and Upgrading of Synfuels from Coal and Oil Shales by Advanced Catalytic Processes; Fifth Interim Report, Sept. 1981). The Base case is being updated to incorporate the above revisions.

Based on conversations with PETC and CAER it was agreed that economic projections would be prepared for each of the four advanced concepts to be tested in the first continuous bench scale run (ALC-1) at HTI. The implications of these projections will be discussed at the ALC-1 Run Plan Meeting to be held at HTI on January 19.

A meeting was held in Lexington with Professor Eric Grulke of the University of Kentucky in an effort to define areas where process modeling using the ASPEN simulator might be useful. Grulke was briefed on similar work done by AMOCO/Bechtel and ICRC and given hard copy reference material.

MISCELLANEOUS

After considerable effort, some data from Exxon's second bench scale run for PETC was obtained. For most of the run dispersed moly was the only catalyst used. A quick review of the run data indicates that both a higher hydrogen treat gas rate and a higher moly catalyst makeup rate (100 vs 50 ppm) increased distillate yield. However, increasing the moly catalyst makeup rate to 750 ppm from 100 ppm had no effect on distillate yield.

Very truly yours,

A handwritten signature in black ink, appearing to read "Michael Peluso", with a long horizontal flourish extending to the right.

Michael Peluso
LDP Associates

cc: F. Derbyshire @ CAER
R. Anderson @ CAER
R. Winschel @ CONSOL
T. Lee @ HTI